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Characterization of Si particles and their effects on and recrystallization in a nanostructured cold rolled Al-1%Si alloy

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Abstract. An Al-1.08 vol.%Si alloy was cold rolled to a reduction of 98% ($\varepsilon_{vM} = 4.5$) and then annealed at different temperatures up to 210°C (0.52 Tm) for different times. The deformed structure is characterized by a nanoscale lamellar structure with the presence of Si particles of coarse (> 1μm), medium (100 nm – 1 μm) and fine (< 100 nm) sizes in the microstructure. Deformation zones are formed around the coarse Si particles and the boundary spacing is finer in the deformation zone than in the matrix. The medium Si particles have little effect on the morphology and boundary spacing. The fine Si particles are aligned along the lamellar boundaries indicating a stabilizing effect on the structural refinement during cold rolling. After annealing, enhanced recovery occurs in the deformation zones around the coarse Si particles. However the reduction in stored energy during recovery and the pinning effect of fine Si particles on the boundary migration prevent the advantage of particle stimulated nucleation (PSN) of coarse Si particles in the nanoscale lamellar structure. This study also demonstrates an important effect of the fine particles in delaying both recovery and recrystallization processes. This effect diminishes with increasing annealing temperature and coarsening the fine particles especially at triple junctions.

1. Introduction

The effect of second phase particles on the deformation microstructure and recrystallization of Al alloys has been extensively studied in the literature [1-7]. It is generally observed that the recrystallization kinetics in alloys containing large particles (typically with sizes > 1 μm) is accelerated while that in alloys containing fine particles is retarded. In the case of containing both coarse and fine particles, the overall kinetics of recrystallization could be retarded or accelerated depending on the relative strength of the effects from the two types of particles [3].

In a recent study [8], a nanostructured Al-1%Si (or Al-1.08% vol.%Si) alloy with an ultrahigh purity Al matrix (99.9996%) was produced by heavy cold rolling to 98% thickness reduction ($\varepsilon_{vM} = 4.5$). The nanostructured Al-1%Si alloy is characterized by a lamellar structure with an average lamellar boundary spacing of 230 nm, a fine dispersion of nanosized Si particles and a certain amount of large Si particles [8]. The Si particles of different sizes are expected to affect the structural and textural evolution during deformation and annealing of the nanostructured alloy. In this study, this effect has been analyzed separately for coarse and fine particles including their combined effect.

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2. Material and experimental procedures

An Al-1%Si alloy ingot was cast of ultrahigh purity (99.9996%) Al and high purity Si. The ingot was hot deformed into a thick plate before being cold-rolled to produce a nanostructured sheet. A slab of dimensions of 300×300×50mm^3 was cut from the hot deformed plate and cold-rolled to a thickness reduction of 98% ($\varepsilon_{vM} = 4.5$). After each rolling pass, the sample was quenched into liquid-nitrogen-cooled alcohol. The rolled sample was annealed at temperatures of 100 – 210°C for different times to investigate the annealing effect on recovery and initiation of recrystallization.

The microstructure was characterized by means of scanning electron microscopy (SEM), electron backscatter diffraction (EBSD) and transmission electron microscopy (TEM) from the longitudinal plane (containing the normal direction and the rolling direction, RD-ND). The SEM/EBSD characterization was done on an FEI Nova 400 field emission gun scanning electron microscope (FEG-SEM). The SEM samples were polished in a 1:9 (vol. fraction) $HClO_4$: C_2H_5COOH solution at −20°C and 20V for 60 seconds. The electron channelling contrast (ECC) technique was used to observe the microstructure. The samples for EBSD were prepared using a Leica EM TIC 3C system. The TEM foils were prepared by a twin jet electropolishing method and examined with a JEOL 2000FX electron microscope operating at 120kV. An online Kikuchi-line analysis system installed in the microscope was used for crystallographic orientation measurements.

3. Results and discussion

3.1 Microstructure and Si particles in the deformed state

The microstructure of the Al matrix is characterized by a fine scale lamellar structure where the lamellar boundaries are low angle dislocation bounadries and high angle grain boundaries [8]. The fraction of high angle bounadries was measured to be 58.4% [9]. The addition of 1%Si is present as particles of various sizes as the soluability of Si in Al is almost zero at room temperature. The Si particles are separated into three goups depending on their sizes: coarse (>1 μm), medium (100 nm – 1 μm) and fine (<100 nm). Figs. 1-3 show examples of observations for coarse-, medium- and fine-sized Si particles. Quantitative measurements of size and volume fraction are listed in Table 1. Some of the large Si particles observed as clusters of medium sized particles are believed to be the result of breaking during cold rolling of large Si particles formed during casting (solidfication) [10]. The majority of the medium sized Si particles are 300 nm to 1 μm. The fine Si particles show a narrow distribution from 5 nm to 30 nm wih an average of 22 nm.

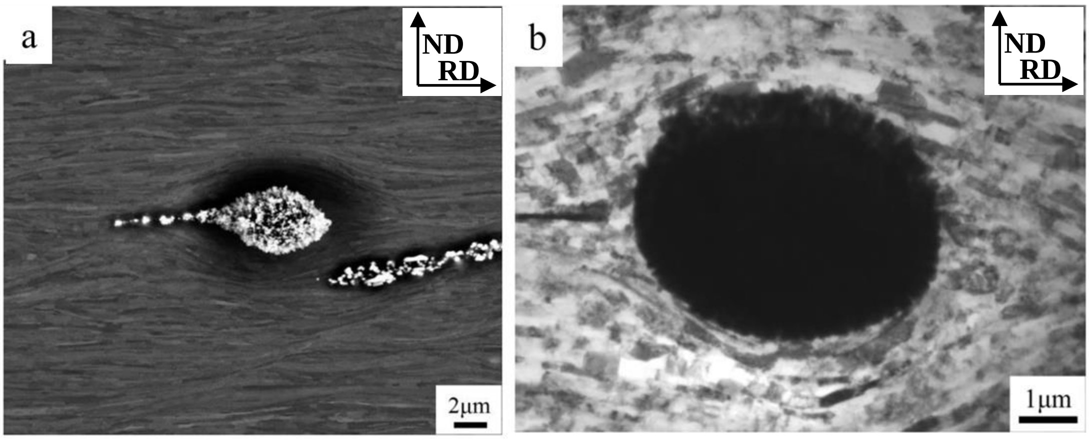

Figure 1. (a) SEM ECC image and (b) TEM image of large a Si particle in a 98% cold rolled Al-1%Si alloy showing the turbulent flow of a aluminum around the particles.

From figure. 1b it is seen that deformation zones of lentical shape are formed around large Si particles where the lamellar boundary spacings becomes finer when approching the particle. However, figure 2 shows that Si particles of medium sizes have little effect on the lamelalr morpholgy of the

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matrix structure and there are no clear deformation zones around the particles. For the fine particles, figure 3, they align along the lamellar boundaries and are also present in the volumes between boundaries. The alignment of fine Si at the lamellar boundaries indicates that they are fomeed during the cold rolling process. The crucial role played by these fine Si particles in producing a nanoscale structure by cold rolling has been discussed in [9, 10].

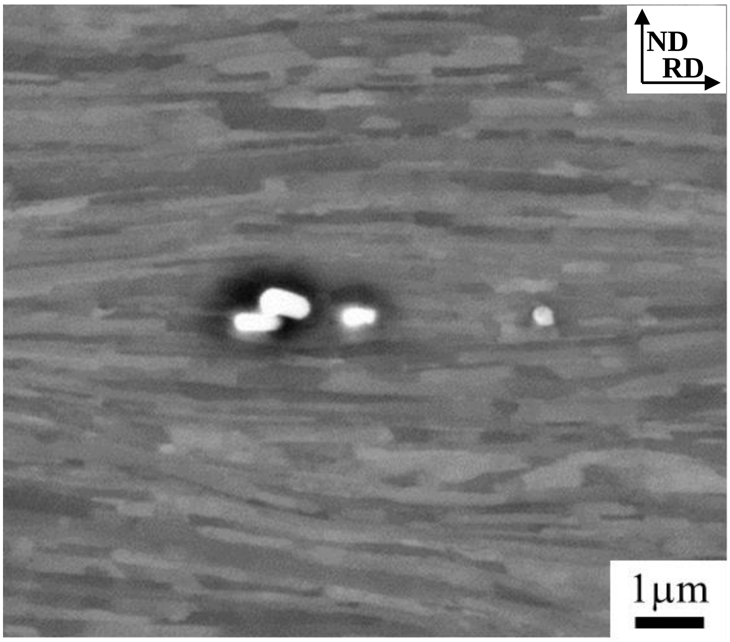


Figure 2. SEM ECC image showing the medium-sized Si particles in a 98% cold rolled Al-1%Si alloy.

To examine the effect of the large Si particles on the deformed structure, the lamellar boundary spacing was measured separatedly for the deformed zones around the large Si particles and for the matrix regions away from the large Si particles. The spacing was measured in the ND i.e. perpendicular to the lamellae and at the particles the spacings between five layers of lamellar structure was analyzed. The measured results are shown in figure 4. The average lamellar boundary spacing in the deformation zones is 180 nm (figure 4a), which is evidently finer than the average value 230 nm measured for the matrix regions (figure 4b).

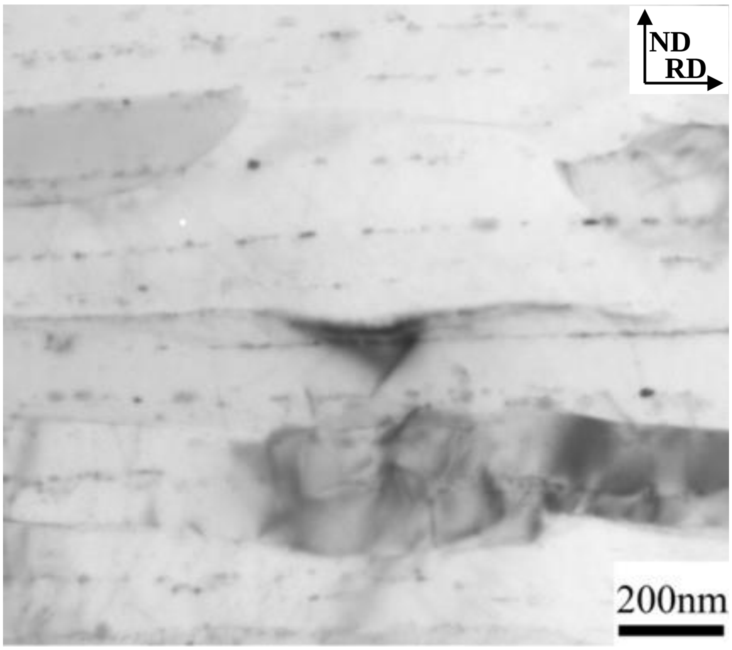


Figure 3. TEM image showing fine Si particles aligned along the lamellar boundaries in a 98% cold rolled Al-1%Si alloy.

Table 1. Size and volume fraction of Si particles in 98% cold rolled Al-1.08 vol.%Si alloy

Particle type	Volume fraction (%)	Average diameter
Coarse (> 1 μm)	0.27	6.80 μm
Medium (100 nm – 1 μm)	0.26	0.65 μm
Fine (100 nm)	0.55	21.5 nm

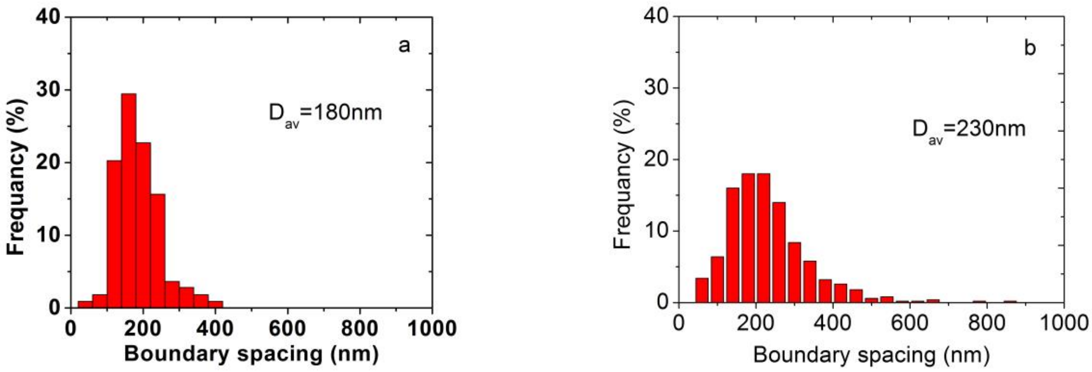


Figure 4. Distribution of lamellar boundary spacing measured separately for (a) the deformation zone around the large Si particles and (b) for the matrix region away from the large Si particles.

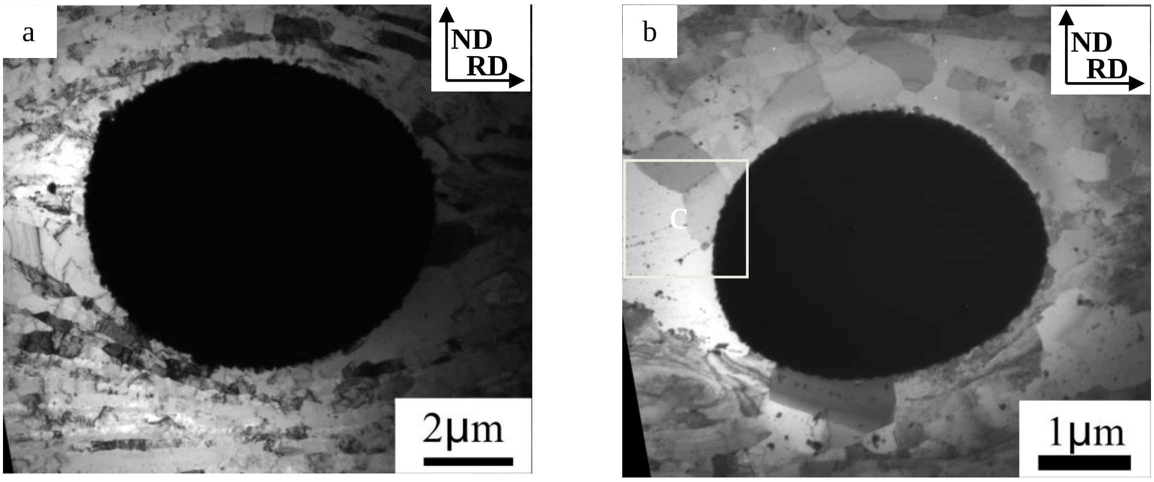
3.2 Recovery of the deformation zone around large Si particles

After recovery annealing, particular attention was paid to obseving microstructural changes in the deformation zones around large Si particles. Figure 5 shows two examples of such observations. Figure 5a shows no obvious change in the lemallar morphology in both the deformation zone and the matrix region after annealing for 1 hour at 150℃ as compared with the deformed state (figure 1b). However, quantification of lamellar bounadry sapcings showed that structural coarsenng has taken place by triple junction motion as observed in aluminum of commecial purity [11, 12]. Enhanced recovery in the deformatin zone around the large Si particle is seen after annealing for 1 hour at 200℃, figure 5b-c. The original fine lamellar structures in the deformed state are now changed to more equiaxed suggrains that have sizes larger than the spacing of lamellar boundaries in the surrouding matrix. Coarsening of fine Si particles may also been seen from figure 5c, but the majority of the fine Si particles are still of sizes less than 50 nm, giving rise to an average of 35 nm. Note that the subgrain sizes around the large Si particles are still in the submicrometre regime, which could be due to the pinning effect of the fine particles present in the matrix as shown in figure 5c. The enhaced recovery of the deformation zones around large Si particles reduced the local stored energy and the pinning effect of fine Si in the same regions (figure 5c) may retard the nuclation proecss at the large Si particles.

3.3 Nucleation and the orientation of nuclei

SEM/ECC observations over large areas for the sample annealed for 1 hour at 200℃ revealed that nuclei or new grains of a few micrometre sizes have been formed, as shown in figure 6a. Some of the nuclei were indeed found to relate to the large Si particles. However, many other nuclei observed seem to have nothing to do with the large Si particles. To evalute statistically whether there is a relation between the formation of recrystallized grains and the large Si particles, several samples were annealed for different periods of time at a slightly higher temperature 210℃and examined by SEM/ECC. An example of such examinations is shown in figure 7b where there more and larger but still isolated recrystallized grains are seen. Again it is seen that the recrystallized grains are not necessarily related to the large Si particles. It is concluded that there is no advantage of particle stimulated nucleation (PSN) [13] at the large Si particles during annealing of the nanostructured Al-1% Si alloy.

The orientations of nuclei formed at the large Si particles and in the matrix were measured by EBSD in a sample that was annealed for 20 minutes at 210℃. More than 70 nuclei were analyzed for each case. Figure 7 shows that {111} pole figure of the orientations for all measured nuclei. The majoiry of the nuclei observed have ramdom orientaions irrespective of whether they formed at the large Si particles or in the matrix. Recently the textural evolution was investigated for the same nanostructured Al-1% alloy until the recrystallization was complete. It was found that the deformed state has a typical rolling texture which is replaced by a rather random texture after the recrystallization is complete [14]. The rather radom orientations of nuclei (figure 7) may explain the formation of the random recrystallization texture. However, clearly this is not particularly related to the effect of large Si paricles in stimulating the formation of nuclei of random orientations, rather the characteristic fine scale lamellar structure and the combined effect of fine and coarse particles on the random nucleation texture may play an important role.



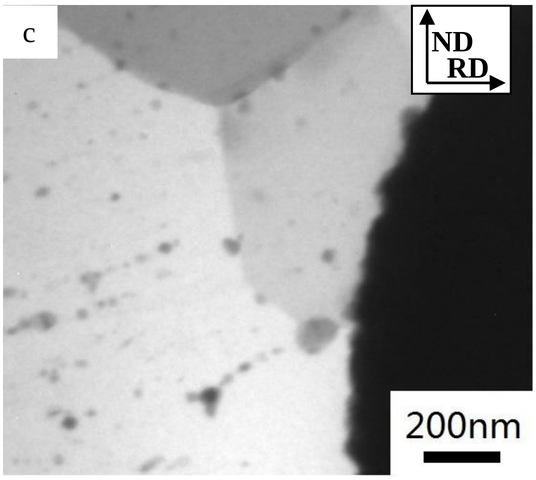


Figure 5. Recovery of the microstructure around large particles after annealing for 1h at different temperatures. (a) 150℃, (b) 200℃, c) Magnified image of the region framed in (b)

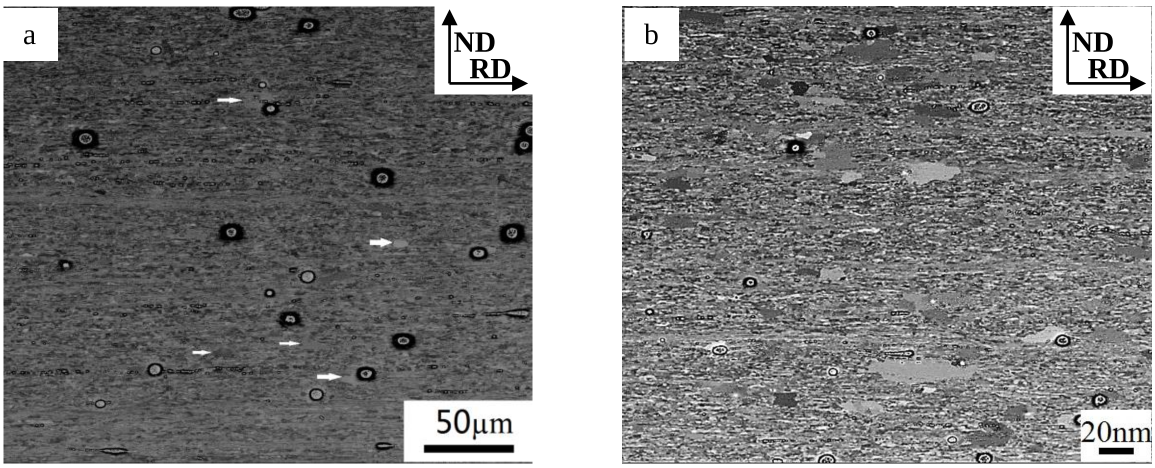


Figure 6. Nucleation at large Si particles and in the matrix observed in samples annealed (a) at 200℃ for 1 hour and (b) at 210℃ for 30 minutes

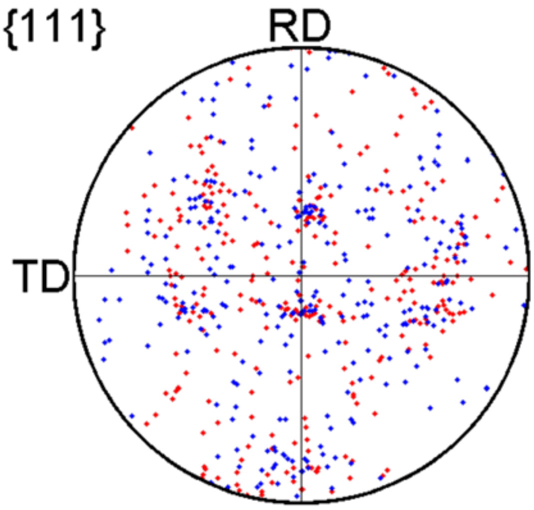


Figure 7. {111} pole figure of the recrystallized grains in Al-Si alloy annealed at 210℃ for 20min. Red points represent nuclei related to the large Si particles and blue points represent the nuclei that are not related to the large Si particles

4. Concluding remarks

The deformation, recovery and recrystallization behaviors have been studied in super-pure Al containing Si particles in the size range from tens of nanometres to several micrometres. The alloy has been cold rolled to 98% reduction (ε_{vM} = 4.5) and afterwards annealed at 150C° (0.45 Tm), 200°C (0.51 Tm) and 210°C (0.52 Tm) to study the recovery and initiation of recrystallization. The cold deformed structure is on the nanometre scale and contains a significant fraction of deformation induced high angle boundaries (>15°). The structure is homogeneous with the exception of regions at and near the large Si particles. The stress and strain in these regions are larger than in the Al matrix, they recover rapidly and PSN is observed. However PSN is not dominating as it competes with abundant nucleation in the Al matrix. It follows that the presence of the fine Si particles is of equal or large importance. The fine Si particles homogenize the deformed microstructure and they retard significant recovery and initiation of recrystallization due to the pinning of dislocation boundaries and high angle boundaries. The presence of fine particles both in the deformed and annealed states may therefore not only increase the thermal stability of the deformed microstructure but also be the cause of the uniform recrystallized grain structure at 250°C and above and the formation of a weak recrystallization texture.

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